

20. (Amended) The method of claim 13 wherein said heat-treating step is carried out at 300-1500°C over a period of 0.1-24 hours and wherein the coating material is selected from the group consisting of indium tin oxide, silicon dioxide, magnesium oxide, sodium phosphate, yttrium-europium oxide, and mixtures thereof; and the precursor solution includes a precursor selected from the group consisting of indium methyl (trimethyl) acetyl acetate, tin isopropoxide, tetraethyl orthosilicate, magnesium nitrate, yttrium chloride, europium chloride and mixtures thereof.

---

Remarks/Arguments

Reconsideration and allowance of claims 1-8 and 10-20 is requested for reasons and comments presented below.

The Interview held on Tuesday, July 30, 2002, at 10 AM, is hereby acknowledged with appreciation when, inter alia, the PTO objections and rejections were discussed. Representing US PTO was the Patent Examiner Michael Cleveland and representing NRL, the federal Applicant, were the inventors Jasbinder Sanghera, Shyam Bayya, Guillermo Villalobos and the attorney George Kap.

Claims 1, 3 -8 and 10-20 remain in this application. Claims 2 and 9 have been canceled and claims 1,3,4,7,8,10,11,13,14,15 and 20 have been amended.

In response to paragraph 1 of the PTO office action, the lengthy specification has been checked and a number of errors have been corrected, as noted above.

In response to paragraph 2, antecedent basis for the claimed

velocities of 0.1-1000 cm/s appears on p. 14, line 6; for heat treating temperatures of 50-200°C, antecedent basis appears on p. 14, line 21; for heat treating times of 0.1-24 hours, antecedent basis appears on p. 15, line 1; and for the phrase "to improve integrity of the coating material," antecedent basis appears on p. 14, line 16. Claims 7 and 8 have been amended to coincide with the disclosure of the parameters in the specification.

In response to paragraph 3, formal drawings were filed in PTO on Sept. 28, 2002.

In response to paragraph 5, claims 1-20 were rejected on the second paragraph of 35 USC 112 as being indefinite in defining removing volatile matter. In reference to claims 1-3 and 13-15, the question has been raised with respect to removal of volatile matter. As noted at the Interview, the volatile matter is removed not only during drying but also during heat treatment and at any time temperature is high enough to drive-off volatile components. As to the scope of the "coating material," it is exclusive of the precursor, as noted in part (c) of claim 1 where it is expressly stated that the coating material is formed from the precursor.

In line 8 on p. 3 of the office action, the Examiner has incorrectly noted that the precursor is heated and is thus decomposed. It is correct to state that the precursor is converted to the coating.

In reference to claims 4-5 and 16-17 noted in paragraph 5 of the office action, the Examiner contends that dilution ratio and particle velocity are unclear. Applicant is amenable to any reasonable proposal

by the Examiner in order to overcome this rejection. In reference to claims 3 and 10, claim 3 has been amended to include any specie of precursor in claim 10 and it is believed that this overcomes the rejection. In reference to claim 13, amendment and discussion thereof at the Interview is believed to have overcome the rejection. Dependency of claim 14 has been corrected.

In reference to paragraph 7 of the office action, as discussed at the Interview, the precursor is not prevented from contacting the particles.

In reference to paragraph 8 of the office action, the Examiner contends that there is no guidance in the specification for formation of other coatings than oxides. Formation of other coatings than oxides from precursors is well known and is prior art. The claims herein are directed to a coating method which the inventors consider their invention. The prior art teaches making organic and inorganic solutions and mixtures thereof. The prior art also teaches that when the solvent is removed, reaction/precipitation takes place.

In reference to paragraph 9 of the office action, as discussed at the Interview, sodium phosphate can be a precursor and the coating. In claim 10, sodium phosphate is claimed as the precursor and in claim 20 it is claimed as the coating. The dual role of sodium phosphate is made possible by its dissolution into sodium and phosphate ions and recombination thereof into the compound sodium phosphate.

In reference to paragraph 10, with deletion of claim 9, issues pertaining thereto are moot.

In reference to paragraph 11, it is believed that objections to claims 10,11,15 and 20 have been overcome by making appropriate corrections in the claims with respect to "acetyl," "ZnS:Ag,Cl" and "hydrates"

In paragraph 13, claims 1-2 and 13-14 were rejected on 35 USC 102(b) as being anticipated by the Peterson reference. In this connection, as the side-by-side comparative color pictorial chart of the Peterson reference (also of the Chau reference) and the NRL Method claimed herein demonstrates, a copy of which was given to the Examiner during the Interview, there is a crucial difference between the NRL method claimed herein and the Petersen's method. The crucial difference resides in the fact that in the NRL method, particles are formed into droplets surrounded by the precursor solution whereas in Peterson, a particle has a coating thereon with the precursor solution surrounding the coated particle. On the pictorial chart, our droplet, which can contain more than one particle (blue), is enveloped by a precursor solution (yellow) and devoid of the coating (red) whereas in Peterson, a particle is already shown as having the coating (red) on its surface with the precursor solution (yellow) surrounding the coated particle. The crucial difference of delaying precipitation of the coating material (red) is believed to be responsible for phosphors which meet the standard of operating under accelerating voltages of 50-10,000 volts without losing more than 50% of the original brightness while operating continuously for in excess of 10,000 hours. The prior art phosphors do not meet the standard. Meeting the standard is consistent

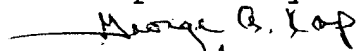
with the objective of the herein-claimed invention.

Claims 1 and 13 have been amended and now recite that the precursor is not precipitated on the particles until after spaying. With amendment of the claims, it is believed that the rejection is no longer applicable.

In reference to paragraphs 16,17 and 18, various claims are rejected under 35 USC 103(a) as being unpatentable over a combination of references wherein the Petersen reference is the prime reference. It is believed that the Petersen reference, modified by the secondary references, does not render obvious the method claimed herein wherein the precursors are not converted to the coating until after spraying or atomizing the particles into droplets.

Attached hereto is a marked-up version of the change's made to the specification and claims by the current amendment. The attached is captioned "Version with markings to show changes made."

Respectfully submitted,

  
Reg. No. 22,898 for

John J. Karasek  
Reg. No. 36,182  
Attorney for Applicants

Prepared by George A. Kap  
Reg. No. 22,898  
Tele.: (703)-404-1555

VERSION WITH MARKINGS TO SHOW CHANGES MADE

In the specification :

Paragraph at line 14, on p. 4, has been amended as follows:

Electrically conducting, non-conducting, luminescent and other coatings can be used to isolate the particle from its surroundings and thereby protect the particle from degradation or reaction with its environment. The coating can also be used to protect the environment from species evolving from the particle. The primary use of this type of coating is to protect phosphors in field emission display devices. Phosphors degrade as a result of electron bombardment and the resulting electrical charging and heating. The coating serves to encapsulate the phosphor and, if it is a conductive coating, to conduct electrical charge away from [form] the particle surface. If the coating is luminescent, one or more of such coatings can be placed on a particle to change the light spectrum of the system. This can be used in emissive displays and in solid state lighting devices.

Paragraph at line 3, on p. 6, has been amended as follows:

An object of this invention is a coating method for particles which provides greater coating uniformity, greatly decreased agglomeration of the particles and greater coating continuity compared [compared] to coatings prepared by other methods.

Paragraph at line 3, on p. 9, has been amended as follows:

In order to fully coat the particles or provide complete hermeticity, it may be necessary to coat the particles more than once. Also, particles made from [of] the coating material, can also be

present since volume of the coating material on a particle is unexpectedly high. To demonstrate the unexpectedly high volume of the coating material on a coated particle, a 90 nm thick coating on a 5-micron particle is equivalent to about a micron particle of the coating material. Particles of the coating material, as well as uncoated particles, also decrease efficiency and brightness of a batch of coated particles containing a preponderance of coated particles where the coating material is different from the particles and a lesser amount of uncoated or partially coated particles and particles of the coating material. Particles made from [of] the coating material are significantly reduced by this method [method].

Paragraph at line 10, on p. 14, has been amended as follows:

Reaction of the precursor component(s) may take place wholly or partially when the precursor solution is sprayed [formed or] in the zones which are [is] typically maintained [maintain] at an elevated temperature.

Paragraph at line 7, on p. 15, has been amended as follows:

The coating thickness on the particles can be varied, inter alia, by adjusting dilutions of the coating solution and/or by adjusting frequency of the atomizer, if an ultrasonic atomizer is used. Although a thicker coating will provide better protection to the particles, generally speaking, thick coatings cost more because more of the coating material is used and function of the particles may be jeopardized. For purposes herein, it has been found that coating thickness in the range of 1-1000 nm, more typically 2-200 nm, is

suitable.

Paragraph at line 7, on p. 17, has been amended as follows:

In this case, a 90 nm (15wt%) indium tin oxide coating on a Nichia ZnS:Ag,Cl [ZnS:Ag.Cl] phosphor particles composed of a mixture of particles of 1-7 microns in diameter and agglomerates 3-9 microns in diameter. A precursor solution was made by mixing in 250 ml of isopropyl alcohol, 1.1 g indium methyl (trimethyl) acetyl acetate and 0.054 g tin isopropoxide. Since the indium and tin alkoxides are not stable in the presence of water, the reaction was carried out in isopropanol that was previously distilled in the presence magnesium to remove any dissolved water from the solvent.

Paragraph at line 13, on p. 18, has been amended as follows:

A stock solution was made by mixing 0.08ml tetraethyl orthosilicate [orthosilicate)], 30 ml ethanol, 0.2 ml water and 0.62 ml hydrochloric acid. One gram of the same phosphor particles as in Ex. 1 was mixed with 2.1 m. of the stock solution and 600ml ethanol. The slurry, at room temperature and at pH of 4.2, was sprayed into a drying zone maintained, as in Ex.1, at 350°C and heat-treated at 450°C for 2 hours. X-ray diffraction of the coated powder showed the presence of ZnS and a broad amorphous hump from SiO<sub>2</sub>. Scanning electron microscope investigation showed the presence of a coating on the particles while energy dispersive x-ray analysis showed the presence of Zn, S, Si and O. Immersing the coated and un-coated phosphor particles in 0.1 molar and 12 molar hydrochloric acid was used to determine continuity of the coating. After 10 minutes, the un-coated phosphor in 0.1 molar



hydrochloric acid showed evidence of acid attack on its surface while the coated sample did not show any damage. The un-coated sample completely dissolved after immersion for 2 minutes in 12 molar hydrochloric acid whereas the coated sample was present after 5 minutes in 12 molar hydrochloric acid.

Paragraph at line 16, on p. 20, has been amended as follows:

This example details the steps to make yttrium-europium oxide luminescent coatings on the ZnS:Ag,Cl [ ZnS:Ag.Cl] phosphor particles.

**In the claims :**

Claim 9 has been canceled and claims 1,3,4,7,8,10,11,13,14,15 and 20 have been amended as follows:

1. (Amended) A method for coating solid particles comprising the steps of

(a) adding solid particles to a liquid coating solution to form a liquid coating slurry containing a coating precursor, solvent for the precursor and the solid particles dispersed therein whereby the precursor is not precipitated until after spraying,

(b) spraying the coating slurry to form droplets containing at least one particle,

(c) passing the droplets through a zone where the droplets are dried and form dry coated particles wherein the coating material is formed from the precursor, and

(d) heat treating the coating material on the particles to remove volatile matter from the coating material.

3. (Amended) The method of claim 1 [2] wherein temperature in

the zone is elevated and the heat treatment of the coated particles is conducted at a temperature above the elevated temperature in the zone, and the precursor is selected from the group consisting of alkoxides, nitrates, sulfates, acetates, hydroxides, hydrates, chlorides, other precursors that can be dissolved in aqueous or non-aqueous liquids, and mixtures thereof.

4. (Amended) The method of claim 3 wherein the particles are less than about 100 microns in diameter, dilution ratio in the coating slurry of milliliters of coating solution per gram of phosphor particles [solids/liquids] is 100-5000, thickness of the coating material on the particles is 1-1000 nm, velocity of the droplets in the zone is 0.1-1000 [100] cm/sec and residence of the droplets in the zone is from instantaneous to a fraction of a minute.

7. (Amended) The method of claim 6 wherein the residence time of the droplets through the zone is 1-5 seconds, the particles are phosphor particles, and said heat treating step is carried out at 50 [200]-2000°C over a period of 0.01-48 [40] hours.

8. (Amended) The method of claim 7 wherein said heat-treating step is carried out at 300-1500°C over a period of 0.1-24 [5] hours.

10. (Amended) The method of claim 8 wherein the coating material is selected from the group consisting of indium tin oxide, silicon dioxide, magnesium oxide, sodium phosphate, yttrium-europium oxide, and mixtures thereof; and the precursor is selected from the group consisting of indium methyl (trimethyl) acetyl [acetyl] acetate, tin isopropoxide, tetraethyl orthosilicate, magnesium nitrate, yttrium

chloride, europium chloride, sodium phosphate and mixtures thereof.

11. (Amended) The method of claim 10 wherein the particles are ZnS:Ag,Cl [ZnS:Ag.Cl] phosphor particles.

13. (Amended) A method comprising the steps of

(a) preparing a liquid precursor solution by dissolving a coating precursor in a liquid precursor solvent;

(b) mixing the precursor solution with a diluent, that is miscible with the precursor solvent, to form a liquid coating solution;

(c) adding with mixing solid particles to the coating solution to form a liquid coating slurry containing the coating precursor dissolved in the coating solution and the solid particles dispersed therein whereby the precursor is not precipitated until after spraying;

(d) spraying the coating slurry to form droplets containing at least one particle;

(e) passing the droplets through a zone where the droplets are dried and form dry particles coated with a coating material formed from the precursor[(s)] solution;

(f) heat-treating the coating material on the particles to remove volatile matter on the coating material and to convert the coating material from electrically non-conducting amorphous to electrically conducting crystalline and/or to improve integrity of the coating material.

14. (Amended) The method of claim 13 [123] wherein condition of the coating slurry is such that no coating material is deposited on

the particles prior to said spraying step.

15. (Amended) The method of claim 13 wherein temperature in the zone is elevated and the heat treatment of the coated particles is conducted at a temperature above the elevated temperature in the zone, and the precursor(s) is selected from the group consisting of alkoxides, nitrates, sulfates, acetates, hydroxides, hydrates [hydrides], chlorides, other precursors that can be dissolved in aqueous or non-aqueous liquids and mixtures thereof.

20. (Amended) The method of claim 13 wherein said heat-treating step is carried out at 300-1500°C over a period of 0.1-24 hours and wherein the coating material is selected from the group consisting of indium tin oxide, silicon dioxide, magnesium oxide, sodium phosphate, yttrium-europium oxide, and mixtures thereof; and the precursor [ (s) ] solution [is (are)] includes a precursor selected from the group consisting of indium methyl (trimethyl) acetyl [acetyl] acetate, tin isopropoxide, tetraethyl orthosilicate, magnesium nitrate, yttrium chloride, europium chloride and mixtures thereof.